To: Zawodny, Peggy[Zawodny.Peggy@epa.gov]
From: Warner, Sue
Sent: Mon 1/27/2014 1:12:37 PM
Subject: FW: purge conditions

From: Rose, Donna [mailto:dlrose@usgs.gov]
Sent: Saturday, January 25, 2014 4:50 PM
To: Warner, Sue
Cc: Donna Rose; Lucinda Murtagh; William Foreman; Duane Wydoski; Caporale, Cynthia
Subject: Re: purge conditions

Hi Sue

I ran both the crude MCHM and the reference standard for 4-HCHM from TCI. Both materials have a small peak just past toluene (14.064 min) that is being identified as a cis or trans isomer of 1,4-dimethylcyclohexane using the NIST library. The spectra has some of the same ions (55 and 97 are the biggest ions) as the 4-MHCM, but different abundances, and elute almost 10 minutes later. The 4-HCHM isomers are eluting between 1-octanol and DBCP on my system. I'm wondering if maybe the peak you were seeing on your purge and trap system is 1,4-dimethylcylcohexane, since you mentioned the spectra didn't quite match the NIST spectra, only one peak was present, and the detection level was in the ppm range. I'm guessing that maybe the 4-MHCM didn't have time to elute off of the column, depending on your GC oven temp program.

I loaded a calibration curve and some reagent water and surface water spikes to run over the weekend. I'll be loading samples on Monday so I hope everything runs ok.

Donna

On Fri, Jan 24, 2014 at 3:55 PM, Caporale, Cynthia <Caporale.Cynthia@epa.gov> wrote:

Donna and Bill,

That is good news and thank you very much for sharing your operating conditions. If you run the actual samples using full scan maybe you can see if the PPH compounds show up. We are testing PPH by P&T on Monday.

Cindy

Cynthia Caporale, Chief
OASQA Laboratory Branch
U.S. EPA Region III
Environmental Science Center
Fort Meade, MD
(410) 305-2732
Fax: (410) 305-3095

From: Rose, Donna [mailto:dlrose@usgs.gov]
Sent: Friday, January 24, 2014 4:55 PM
To: Foreman, William
Cc: Warner, Sue; Caporale, Cynthia; Zawodny, Peggy; Donna Rose; John Zogorski; Duane Wydoski; Lucinda Murtagh; David Reppert
Subject: Re: purge conditions

Typo on the compound name- should be 4-methyl-1-cyclohexanemethanol.

Donna

On Fri, Jan 24, 2014 at 2:39 PM, Rose, Donna <dlrose@usgs.gov> wrote:

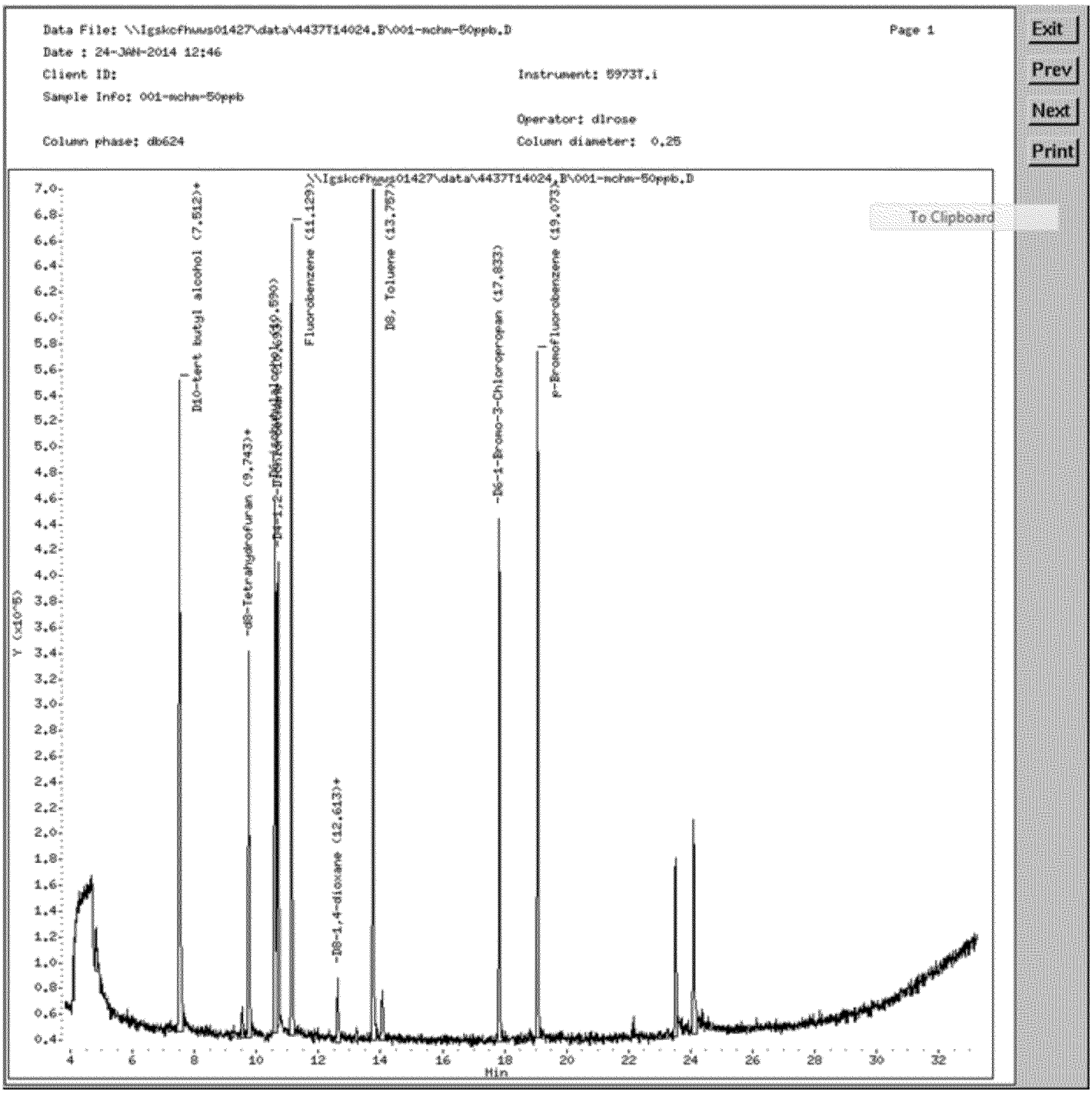
Hi Everyone

I got the standard in for 4-methyl-1-cyclohexanemethane and ran a 50 ug/L standard on my system. The mass spec is in full scan/sim simultanous mode. The operating conditions are listed in the attached excel file.

I got two peaks, one at 23.514 minutes and one at 24.094 minutes. Both matched the NIST reference spectra beautifully. I don't know which peak is the cis isomer and which is the trans isomer. TCI had a purity of 99.8% for the total isomer purity. I left a message with technical support to see if they had additional information on the percentages of each isomer.

The response is beautiful in the full scan mode. The second peak is slightly bigger than the first but not by much. The two peaks at the end of the chromatogram are the MCHM isomers.

full scan data file



Donna Rose, Chemist

U. S. Geological Survey

303-236-3283

On Thu, Jan 23, 2014 at 3:19 PM, Foreman, William <wforeman@usgs.gov> wrote:

Sue,

Thanks for the info and call. We'll let you know how it goes for us.

Bill

On Thu, Jan 23, 2014 at 2:36 PM, Warner, Sue <Warner.Sue@epa.gov> wrote:

Recommended Operating Conditions for Purge and Trap Apparatus for Volatiles in Soil Analysis (Low Level) Using the Archon Autosampler

Operating Parameter	**Setting**
Sample Type	Soil
Sample Volume	5 or 10 mL (adds 5 or 10 mL: 10 mL for samples and blanks and 5 mL for standards and spikes)
Rinse Volume	25 mL
Number of Rinses	2
Standard 1	Yes
Standard 2	No
Soil Preheat Stir	Yes
Stir	Yes
Syringe Flushes	2
Preheat	Yes
Preheat Temperature	40 °C
Preheat Time	1.5 min.
Purge Time	11.0 min.
Desorb Time	2.0 min

Operational Mode	Remote
Cycle Timer	38.3 min
Aux Time	0.0 min
Purge Gas Pressure	20 psi
Flow	35-40 mL/min

The trap used is a VOCARB 3000.

From: Caporale, Cynthia
Sent: Thursday, January 23, 2014 4:18 PM
To: Warner, Sue; Zawodny, Peggy
Subject: FW: purge conditions

FYI – feel free to speak to the voc analyst directly or if you want a conference call I'd be willing to help set one up.

From: Foreman, William [mailto:wforeman@usgs.gov]
Sent: Thursday, January 23, 2014 4:10 PM
To: Caporale, Cynthia
Cc: Donna Rose; William Foreman
Subject: purge conditions

Hi Cindy,

I'd appreciate your checking with your P&T analyst regarding the following conditions used:

purge temp

purge time

purge volume

type of trap

I've cc'd our lead VOC analyst, Donna Rose. If your analyst has time for a brief call today or

tomorrow, please have them call Donna at 303-236-3283. Or we can set a time and do it as a conference call.

If you happen to have any handy details about the ERT mobile lab's method, that would be helpful.

We greatly appreciate the help/guidance!

Bill

William T. Foreman, Ph.D.

Research Chemist
Methods Research and Development Program
U.S. Geological Survey
National Water Quality Laboratory
P.O. Box 25585
Denver, CO 80225-0585
{For Fedex, delete Box number and add Bldg. 95, Entrance E3}
303-236-3942; FAX: 303-236-3499
email: wforeman@usgs.gov

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Donna L. Rose

Chemist

dlrose@usgs.gov

U.S. Geological Survey

National Water Quality Laboratory

303-236-3283 phone

303-236-3499 fax

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Donna L. Rose

Chemist

dlrose@usgs.gov

U.S. Geological Survey

National Water Quality Laboratory

303-236-3283 phone

303-236-3499 fax

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Donna L. Rose

Chemist

dlrose@usgs.gov

U.S. Geological Survey

National Water Quality Laboratory

303-236-3283 phone

303-236-3499 fax